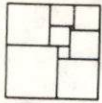


6746



NUS
CORPORATION

992 OLD EAGLE SCHOOL ROAD
SUITE 916
WAYNE, PENNSYLVANIA 19087
(215) 687-9510

February 22, 1984
R-585-7-3-14
68-01-6699

Mr. Harold Byer
U.S. Environmental Protection Agency
Sixth and Walnut Streets
Philadelphia, PA 19106

Subject: Final Field Trip Report
TDD No. F3-8306-20
FMC-Baltimore
Baltimore, Maryland

Dear Mr. Byer:

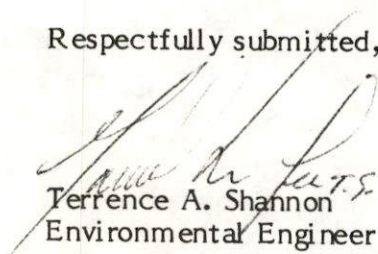
Submitted herewith is a final Field Trip report for the subject project.

Based on our review of available data, we have concluded that EPA should consider the following:

- o 2,3,7,8-TCDD was not detected in any of the 12 samples taken from FMC property.


If you have any questions, please contact me (215) 687-9510.

Respectfully submitted,

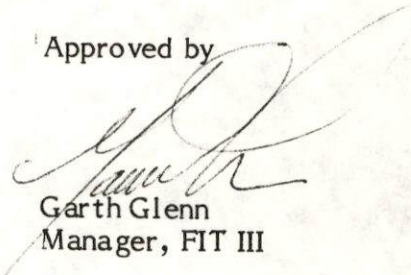

Terrence A. Shannon
Environmental Engineer

Laura A. Boornazian
Air Pollution Specialist

Reviewed by


William Wentworth
Asst. Manager

Approved by


Garth Glenn
Manager, FIT III

TAS/LAB/ldm

R-585-7-3-14
A FIELD TRIP REPORT FOR
FMC BALTIMORE
PREPARED UNDER

TDD NO. F3-8306-20
EPA NO. MD-17
CONTRACT NO. 68-01-6699

FOR THE
HAZARDOUS SITE CONTROL DIVISION
U.S. ENVIRONMENTAL PROTECTION AGENCY

FEBRUARY 22, 1984


NUS CORPORATION
SUPERFUND DIVISION

SUBMITTED BY


TERRENCE SHANNON
ENVIRON. ENGINEER

LAURA A. BOORNAZIAN
AIR POLLUTION SPECIALIST

REVIEWED BY


WILLIAM WENTWORTH
ASST. MANAGER

APPROVED BY


GARTH GLENN
MANAGER, FIT III

TABLE OF CONTENTS

<u>SECTION</u>		<u>PAGE</u>
1.0	INTRODUCTION	1-1
1.1	AUTHORIZATION	1-1
1.2	SCOPE OF WORK	1-1
1.3	SUMMARY	1-1
2.0	FIELD TRIP REPORT	2-1
2.1	SUMMARY	2-1
2.2	PERSONS CONTACTED	2-1
2.2.1	PRIOR TO FIELD TRIP	2-1
2.2.2	AT THE SITE	2-1
2.3	SAMPLE LOG	2-3
2.4	SITE OBSERVATIONS	2-4
2.5	PHOTOGRAPHIC LOG	2-10
3.0	LABORATORY DATA	3-1
3.1	SAMPLE DATA SUMMARY	3-1
3.2	QUALITY ASSURANCE REVIEW	3-2
3.2.1	DIOXIN DATA	3-2
3.3	REANALYSIS OF SAMPLE M-02-13	3-4
<u>APPENDICES</u>		
A	SAMPLE BLENDING PROCEDURE	A-1
B	FMC LETTER (6/29/83) RESPONSE TO EPA-III LETTER OF 6/15/83	B-1
C	MAPS/SKETCHES	C-1
D	Q/A SUPPORT DOCUMENTATION	D-1
E	SAMPLE PAPERWORK	E-1
F	ANALYTICAL RESULTS	F-1

1.0 INTRODUCTION

1.1 Authorization

NUS Corporation performed this work under Environmental Protection Agency Contract No. 68-01-6699. This specific report was prepared in accordance with Technical Directive Document No. F3-8306-20 for the FMC Inc. Baltimore Plant, located in Curtis Bay, MD.

1.2 Scope Of Work

NUS FIT III was tasked to conduct sampling of FMC's Baltimore Plant in Curtis Bay, MD. Samples were analyzed for dioxin (2,3,7,8-TCDD) and priority pollutants. The investigation was conducted by NUS personnel Terrence Shannon, Eugene Dennis, Richard Cromer, Michael Nalipinski, and David Hassrick.

1.3 Summary

In accordance with the listing of FMC's Baltimore plant in the EPA "Dioxin Report", NUS FIT III conducted a sampling program consisting of screening for dioxin (2,3,7,8-TCDD) and organic and inorganic priority pollutants.

NUS FIT III personnel attended a number of meetings in preparation for the investigation. A meeting was held with personnel from the Center for Disease Control (CDC) in Atlanta, Georgia. In order to ensure a sampling program that would complement EPA III's and CDC's needs, discussions were held regarding the type of sampling plans (screening vs. statistical), sample locations (pipes, tanks, dust, etc.), quality assurance/control programs (performance audit, duplicate, and spiked samples), and on-site procedures (homogenization of samples, personnel protection).

In addition, a meeting of the Dioxin Work Group was attended by NUS FIT III personnel, in which administrative and technical details were finalized. Prior to initiating the on-site work, briefings were held with the individual work teams to ensure that all aspects of the investigation were performed in accordance with the newly established protocols for sample preparation and sample/personnel decontamination.

EPA III officials initially contacted FMC, Inc. with a letter requesting information related to the processing and handling of the Tetradifon ("Tediion") noted in the Dioxin report. At that time, a date was arranged for both a preliminary meeting, as well as the actual sampling.

The on-site meeting and investigation took place on June 20, 1983. The preliminary meeting was attended by personnel from NUS, FMC, EPA III, the MD WRA, and the Baltimore Dept. of Health. NUS FIT III personnel initiated the sampling of those locations decided upon by EPA III at the meeting. Samples were obtained from 12 locations on the plant property.

Sample analytical results were received on 7/25/83. The results did not detect 2,3,7,8-TCDD in any of the 12 samples taken from FMC property. However, a QA/QC check of the dioxin results indicated interferences in the results for sample number M-02-13. It should be noted that only 2,3,7,8-TCDD results are presented in this report. Results of sample analysis for priority pollutants will be forthcoming in a separate report.

2.0 FIELD TRIP REPORT

2.1 Summary

Pursuant to the Technical Directive Document #F3-8306-20, site sampling of the FMC, Inc., Baltimore plant was conducted on June 21, 1983. NUS FIT III personnel who participated in the inspection included Terrence Shannon, Eugene Dennis, Richard Cromer, Michael Nalipinski, and David Hassrick (Dioxin Team "B").

The weather during the inspection was overcast, with a temperature of 70° F and winds from 0-5 mph. A slight rainfall of short duration occurred during the inspection.

Samples were obtained from twelve stations at the plant for dioxin and organic/inorganic priority pollutant analyses. Split samples were provided FMC personnel, under chain-of-custody.

2.2 Persons Contacted

2.2.1 Prior to Field Trip

All contacts prior to the on-site work with the facility were made by EPA III's representative, Neil Swanson (3AW23). NUS FIT III personnel did not have any contact with facility personnel until arriving at the site.

2.2.2 At the Site

Upon arriving at the site, a meeting was held with the following:

Peter Schaul
Neil Swanson
EPA, Region III
6th and Walnut St.
Philadelphia, PA 19106
(215) 597-9800

Donald Senovich, FIT Manager
Terrence Shannon, Engineer
NUS Corporation
992 Old Eagle School Road
Wayne, PA 19087
(215) 687-9510

Elkins Dahle, Jr., Director
Charisse Deutch, Inspector
Baltimore Bureau of Industrial Hygiene
111 N. Calvert St., Rm S-219
Baltimore, MD 21202
(301) 396-4477

Darryl Palmer, Environmental Manager
Frank Soleck, Production Manager
Irv Kipnis, Process Laboratory Manager
Chuck Shaheen, Environmental Engineer
FMC Corporation
Agricultural Chemical Group
1701 E. Patapsco Ave., Box 1616
Baltimore, MD 21203
(301) 355-6400

Joseph Stang, Inspector
MD Dept of Health & Mental Hygiene
Office of Environmental Programs
201 W. Preston St., Rm. 2A4
Baltimore, MD 21201
(301) 383-6650

TDD Number 8306-20
EPA Number MD-17

SAMPLE LOG
Dioxin

[illegible]

2.4 Site Observations

FIT III personnel arrived on-site at 0900 on June 20, 1983, to attend a meeting previously arranged by EPA III representative Neil Swanson. Mr. Swanson described the screening nature of the anticipated sampling program and the analyses to be run (2,3,7,8-TCDD and organic/inorganic priority pollutants). A general discussion ensued, concerning the plant's layout and the production history of Tetradifon, the compound of concern to EPA due to its potential for dioxin contamination. The following information was elicited from the FMC personnel present at the meeting.

The total size of the plant is approximately 50 acres, with 20 acres located north of Patapsco Ave. and 30 acres located south of Patapsco Ave. Approximately 350 people are employed at the plant.

Relative to the production of Tetradifon, Building #91 was the location of the compound's pilot plant. Building #91 was reconfigured and is currently the site of "Pounce" (permethrin) production, a chemical used on cotton and tobacco. The equipment used for the Tetradifon production was either decontaminated and used elsewhere in the plant or scrapped and sold. The exact fate of this equipment was unknown.

The semiworks for the commercial production of the Tetradifon was located in Building #52 and was in operation from approximately 1960 through 1970. Building #52 was demolished, date unknown, and is currently the location of the plant's RCRA waste facility. This area is sealed with an asphalt pad.

All product generated by the pilot plant and the semiworks for Tetradifon were drummed and transported by truck. There was no railroad transport, to the best of the FMC personnel's recollection. Likewise, an incinerator constructed on the plant's property in 1968 was not used for the incineration of any Tetradifon production wastes.

Three CERCLA areas of the plant were discussed. One area was located adjacent to the old "waste-pond" area in the plant's southeast quarter. FMC personnel stated that the area, formerly a wetlands, was used for the disposal of aqueous waste from the production of Tetradifon, as well as unknown, miscellaneous materials. The pond was excavated, filled, and a storage facility was constructed on the site. The pond's contents were removed and possibly disposed of at Solley Road landfill. The area is currently capped with a sand/gravel cover. A second CERCLA area, located south of Building #91 and north of Patapsco Ave. was also used for the disposal of unknown, miscellaneous materials. It is the former location of acetic acid production facilities. A third CERCLA area, located approximately 200 feet west of Building #89, was the location of a tank of unknown contents and fate.

The plant has two runoff collection systems. One system, called the plant general system, drains the entire plant except for the 7-OH production area. The general system discharges to a POTW. The collection system for the 7-OH area discharges to the retention basin located on the plant's southern boundary. Effluent from the retention basin is discharged via an NPDES permit to the Patapsco River.

In regard to the technical aspects of the inspection, it was decided that splits of both the dioxin and priority pollutant samples would be provided FMC. Material for the dioxin analysis would be collected in the blender top, homogenized with the blender, and split. Material for the priority pollutant analysis would be collected in a stainless steel bucket, mixed with a stainless steel scoopula, and split. FMC would provide their own glassware for their split samples. Photographs of the sampling would be obtained by FMC, developed, and mailed to the NUS FIT III contact, Terrence Shannon. Finally, FMC personnel would accompany NUS FIT III personnel during all plant surveys and sampling.

Following the discussion, a walk-through of the facility was conducted. See next page.

The southeast section of the plant was inspected, including the retention basin, the fire water basin, the old waste pond area (near the 7-OH control room, Building #80), and a CERCLA-reported inactive fill area. Both the old waste pond area and the CERCLA fill area were covered with what appeared to be a white, 2 to 3 inch-sized gravel, with an underlying area of fine sand, which presumably was underlain by the old waste areas. There were no signs of environmental contamination in those areas.

The inspection team proceeded to the unnamed stream which bordered the eastern portion of the south part of the facility. Recent heavy rains had severely flooded this area. Access to this portion of the facility was via the east gate, which was used at one time by all contractors entering the plant.

The inspection team proceeded to the former location of Building #52, used for production of the potential dioxin contaminated product Tetradifon. The area is currently occupied by a RCRA waste storage facility and is completely covered by asphalt and gravel of the type mentioned earlier. An area of sediment accumulation was observed underneath steps on the northwest side of the area. An open drain was observed in a shallow depression area on the southwest side of the area. A railroad spur, running north/south, was located on the other side of a plant access road located immediately adjacent to the area's western side. Building #34 was located to the west of the area.

The team proceeded to the warehouse area adjacent to the plant's 2nd St. A railroad spur, embedded in a concrete causeway, was located parallel to 2nd St., next to the warehouse. The loading platform for the warehouse area was observed at the western end of the buildings. The railroad bed material could not be ascertained.

The team proceeded to Building #91, located on the north side of Patapsco Ave. The facility, formerly the pilot plant for the Tetradifon product, is currently used for "Pounce" (permethrin) production. An area of soil was observed behind the building, amidst the production plant's waste treatment area. An area of grass covered soil, containing scrub growth and small trees, was located near the building's southeast corner. An asphalt parking lot/driving area bordered the building. The area occupied by the building is bordered on three sides (north-east-west) by three different companies. The interior of the building contained Pounce-related equipment, with the piping displaying fairly recent painting.

The team proceeded to the CERCLA-fill areas located south of Building #91 and north of Patapsco Ave. The area consisted of foundations for former acetic acid production facilities. The area was very overgrown and contained standing water, due to the previous heavy rainfall of June 19, 1983. One specific area was pointed out by Mr. Palmer as an area that FMC knew contained unknown, miscellaneous material. As far as the rest of the area, Mr. Palmer had no information.

A third CERCLA area was pointed out by Mr. Palmer, located northwest of Building #91's parking lot. The area was reportedly the former location of a tank of unknown origin and contents.

Following the completion of the inspection, personnel returned to the conference room for development/discussion of the sampling plan. Upon completion of the discussion, FIT III personnel departed the site at approximately 1330 hours.

FIT III personnel returned to the plant on June 21, 1983, to conduct sampling. Prior to the inspection, FIT III personnel were briefed on FMC safety procedures. Lines of communication, investigation protocols, and sample locations were also discussed. Personnel then proceeded to the north side of the facility (north of Patapsco Ave.). After establishment of the Command Post (CP) near Building #89, sampling was initiated.

Three bore holes were advanced on the northern, eastern, and western boundaries of the old acetic acid production area, which was reported by the plant under CERCLA. There were no signs of environmental contamination. Sample numbers M-02-01, 02, and 03 were obtained.

Sampling personnel then obtained sample number M-02-04 from the bed of a railroad spur which serviced the plant. The spur was located on the plant's northeast quadrant. The material from the railroad bed consisted of a crumbly, black solid, which did not display signs of environmental contamination.

Sample number M-02-05 was obtained from the location of the storage tank, which was also reported by the plant under CERCLA. A duplicate sample, in addition to the split samples, was obtained from this station for QA/QC purposes. There was no sign of environmental contamination.

Sample numbers M-02-06 and M-02-07 were obtained from around Building #91. Sample number M-02-06 was obtained from a grass/soil area located amidst the process works for the permethrin produced at Building #91. There were no signs of obvious environmental contamination.

Sample number M-04-07 was obtained from a small lawn area located in the front of Building #91. A surface soil sample was obtained from this station. There were no signs of environmental contamination.

Sample number M-04-08 consisted of decontamination rinsate (1,1,1-trichloroethane) used to decontaminate the blender tops. This sample was also obtained in accordance with QA/QC requirements.

Upon the completion of the processing for sample number M-02-08, the CP was transferred to the plant's southern side (south of Patapsco Ave.). Sampling on the facility's south side initiated at the inactive fill/waste pond area. Sample numbers M-02-09 and M-02-10 were obtained from the eastern and western boundaries of the area. There were no signs of environmental contamination. To obtain the samples, the top layer of ground and sand was removed, and a shallow stem auger was used to obtain the samples.

An attempt was made to sample the East Gate area (#M-02-11). However, the gravel pack was too dense and a sample could not be obtained.

The sampling personnel proceeded to the former location of Building #51, adjacent to Building #34, to obtain sample numbers M-02-12 and M-02-13. Sample M-02-12 was obtained from an area located near Building #34's southeast corner. Sample M-02-13 was obtained from soil underneath a foundation for current steps located near Building #34's northeast corner. As with sample numbers M-02-09 and M-02-10, the top layers of gravel and sand were removed, then the auger was advanced to refusal. No signs of environmental contamination were noted.

A final sample, intended as a clean field blank, was obtained from a lawn located in front of Building #19. A duplicate sample, in addition to the splits, was obtained for QA/QC purposes.

All samples for dioxin analysis were processed in accordance with Document #C-585-6-3-54 (See Appendix A). Upon completion of the sampling, split samples were provided to FMC personnel under chain-of-custody. All samples were obtained, photographed, processed, documented, packaged, and shipped in accordance with accepted protocols. All solid and liquid wastes generated during the inspection were drummed and removed from site. Upon completion of the breakdown of the CP, FIT III personnel departed the site at 1815 hours.

3.0 LABORATORY DATA

3.1 SAMPLE DATA SUMMARY

**SAMPLE DATA SUMMARY
TARGET COMPOUNDS**

TDD Number F3-8306-20

EPA Number MD-

☒ Organic ☐ Inorganic

2,3,7,8-
TCDD

Site Name Fmc, Baltimore

Date of Sample 6-21-83

Compounds Detected

Sample Number	Sample Description and Location	Phase	Units	2,3,7,8-TCDD														Remarks
M-02-01	Facility, North Side	SOL	ppb	ND														
M-02-02	Facility, North Side	SOL	ppb	ND														
M-02-03	Facility, North Side	SOL	ppb	ND														
M-02-04	Facility, South Side	SOL	ppb	ND														
M-02-05	Facility, North Side	SOL	ppb	ND														
M-02-06	Facility, North Side	SOL	ppb	ND														
M-02-07	Facility, North Side	SOL	ppb	ND														
M-02-08	Facility, North Side	AQ	ppb	ND														
M-02-09	Facility, South Side	SOL	ppb	ND														
M-02-10	Facility, South Side	SOL	ppb	ND														
M-02-12	Facility, South Side	SOL	ppb	ND														
M-02-13	Facility, South Side	SOL	ppb	ND														
M-02-14	Facility, South Side	SOL	ppb	ND														

NOTE: For a review of this data and non-target, tentatively identified compounds, please see the Analytical Quality Assurance section of this report.

3.2 Quality Assurance Review

3.2.1 Dioxin Data: Lab Case: SAS-619C

3.2.1.1 Introduction

The findings offered in this report are based upon a general review of all available dioxin sample data. Blank analysis, surrogate, matrix spike, duplicate, and performance audit results, calibration standards, and isomer separation standards were examined in detail.

3.2.1.2 Qualifiers

It is recommended that this data package be utilized only with a qualifier stating that the initial results for sample M-02-13 did not rule out the presence of 2,3,7,8-TCDD at the required detection limit of one part per billion. Several reanalyses of this sample were performed, and these results are addressed in Section 3.3 of this report.

3.2.1.3 Findings

- o Cleanup options A and D of the Region VII protocol were used in an attempt to eliminate interferences which precluded the determination of any native 2,3,7,8-TCDD and the internal standard in sample M-02-13. The sample was re-extracted and reanalyzed in order to obtain the required detection limit, and results are discussed in a separate Quality Assurance Review (Section 3.3 of this report).
- o One of the two chromatographic columns used in this project did not meet the interim isomer resolution criteria established in Kansas City on July 13, 1983. However, data generated on either column is adequate to rule out the presence of indigenous 2,3,7,8-TCDD. In order to obtain accurate quantitation and confident isomer specificity, the laboratory was directed to reinject all samples having possibly positive results for 2,3,7,8-TCDD on another column which met the criteria. (The only positive samples in this case turned out to be the two performance audit samples and the laboratory matrix spike.)

3.2.1.4 Summary

The attached Quality Assurance Review has revealed chromatographic interferences in sample M-02-13 as the major area of concern. Please see the accompanying Support Documentation Appendix to this report for specifics on this Quality Assurance Review.

Report prepared by Russell J. Sloboda *Russell J. Sloboda* Date: July 25, 1983

3.3 Quality Assurance Review

3.3.1 Reanalysis of Sample M-02-13 Dioxin Data: Lab Case: SAS-619C

3.3.1.1 Introduction

The findings offered in this report are based upon a general review of all available data for three reanalyses of sample M-02-13. The data examined represent an EPA-requested low resolution GC/MS analysis, an FMC-funded/requested high-resolution GC/MS analysis of the same sample, and an FMC-funded/requested low resolution GC/MS analysis of a split portion of this sample. (The original sample was mechanically blended in the field before splitting was performed.) EPA's low resolution GC/MS analysis detected dioxin at 1.04 ug/kg, whereas the other two analyses did not find dioxin. Detection limits were 0.27 ug/kg for the high resolution analysis and 0.20 ug/kg for the FMC low resolution analysis.

3.3.1.2 Qualifiers

It is recommended that this data package be utilized only with the following qualifier statements:

- o The detection limit for the high-resolution analysis was incorrectly reported by the laboratory. The corrected limit is 0.27 ug/kg.
- o Although the existing sample data is insufficient to unambiguously determine the cause of the discrepancy between the positive and non-detected sample results, several pieces of evidence suggest that the one positive result for dioxin may be an artifact of chemical interference(s) which exhibit a response similar to that of dioxin.

3.3.1.3 Findings

- o The error in the reported detection limit arises out of the interpretation of the section of the dioxin protocol which addresses calculation of detection limits. The corrected limit was calculated as 2.5 times the amount represented by the lower level interfering mass areas compared to the corresponding C_{13}^{12} -TCDD mass area. This is different from the reported detection limit, which was calculated using the sum of the two masses 320 and 322, and which yielded a higher result due to a relatively higher interference at mass 322 versus mass 320.
- o The sample contained high level interferences which necessitated additional preparatory effort for all three laboratories. Even after cleanup, multiple interferences containing co-maximizing mass 320 and 322 peaks were observed by all laboratories. In the FMC-funded low resolution GC/MS analysis, one particular interference displayed the correct 320/322 ion ratio, but without the 257 ion or retention time characteristic of dioxin. Another interference contained all three ions 320, 322, and 257, but did not exhibit the correct 320/322 ion ratio or retention time characteristic of dioxin. (In the EPA low-resolution GC/MS analysis, dioxin was identified as a peak having the correct retention time for all three ions, and a 320/322 ion ratio within the acceptable range.)
- o Conversations with several chemists have revealed that interferences have occasionally produced artifactual low-resolution GC/MS results for dioxin. In this case, the high-resolution result should be given greater credibility due to the capability of this method to eliminate artifactual interferences that the low-resolution method cannot distinguish.
- o To be sure, the high-resolution result does not disprove the validity of the one positive low-resolution result, since a different aliquot was taken for each analysis, and the sample might not have been as homogeneous as expected from the field blending. However, even if the positive result is not artifactual, the results from the other two analyses suggest that the average level of dioxin present is less than 1.0 ppb.

Site Name: FMC Baltimore Plant
TDD No.: F3-8306-20

- o Thus, in order to confidently determine if the one positive result is valid or not, the original extract would have to be reanalyzed using a partial scan or high-resolution technique. However, this analysis could be successful only if significant losses of internal standard and surrogate have not occurred as a result of storage and handling of the extract.

The attached Quality Assurance Review has identified an incorrect detection limit and a possible artifactual result due to chemical interference as the major areas of concern. Please see the accompanying Support Documentation Appendix to this report for specifics on this Quality Assurance Review.

Report prepared by Russell J. Sloboda *Russell J. Sloboda* Date: November 18, 1983

**Sampling
Blending Procedure**

C-585-6-3-54

Samplers take sample in 1 qt. stainless steel blender cup.

Blender cup should be filled no more than 3/4 full.

Note: Attempt to avoid placing stones in the blender cup. Samplers should also break up large clumps of soil.

Sample is then returned to blending station.

Blending procedure will commence as follows:

- 1 Pulse blender five (5) times.**
- 2 Invert blender cup several times and shake.**
- 3 Repeat this procedure six (6) times for a total of 30 pulses.**
- 4 Allow the blender to sit for two to five minutes to allow all dust to settle.**

Person who is blending removes right glove to open sample jar, glove is put back on when filling the jar.

Sample will be removed from the blender cup utilizing scoopulas which will be disposed of when the sample jar has been filled.

Right glove is removed for the capping of the jar.

Remove baggie and rubber band and place in designated receptacle.

Sample jar is decontaminated with 1,1,1-trichloroethane if visual contamination is evident.

Sample is then tagged, and processed by the site leader.

Any material remaining in blender cup is disposed of in the waste receptacle.

Blender cup is cleaned with soap and water and scrubbed with brush if necessary.

Blender cup is filled 1/4 to 1/2 full with soapy water and agitated (blended) for 30 seconds.

Cup is then rinsed with distilled water, alcohol, and 1,1,1-TCEa. Allow to drip dry.

Sample cup is ready to receive next sample.

FMC Corporation

Agricultural Chemical Group
1701 East Patapsco Avenue Box 1616
Baltimore Maryland 21203
(301) 355 6400

FMC

June 29, 1983

Mr. Neil Swanson
Environmental Scientist
Waste Enforcement Section
Air and Waste Management Division (3 AW 22)
United States Environmental
Protection Agency
Region III
6th and Walnut Streets
Philadelphia, PA 19106

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

Re: Response to EPA Region III
Inquiry on Possible Formation
of Dioxins

Dear Mr. Swanson:

On June 20, 1983, FMC Corporation's Agricultural Chemical Group plant in Baltimore, Maryland received a letter dated June 15, 1983 from Region III of the U. S. Environmental Protection Agency, in which EPA Region III inquired as to certain information relative to the possible formation of dioxins at FMC's Baltimore plant in the course of manufacturing and handling practices over the years involving various organic chemicals. Among other things, EPA Region III's June 15, 1983 letter requests submission within ten days of receipt of the letter of a written report on the status of discussions with EPA Region III or on other action relative to the matters set forth in the letter. By the present letter, we are providing the report thus requested. In doing so, however, we do not intend to express any opinion as to the applicability of the statutory provisions referenced in the first paragraph of EPA Region III's letter or to any other statutory requirement for such a report. In response to the information requested in Attachment II of EPA Region III's letter, we are in the process of reviewing our files. We will be in a better position to respond further to this request after this review and will be in contact with you by July 10, 1983.

On Monday, June 20, 1983, a meeting was held at the Baltimore plant involving Peter Schaul and yourself from EPA Region III, myself and other FMC representatives, and representatives from your prime contractor (NUS Corporation), the City of Baltimore Health Department, and the State of Maryland Office of Environmental Programs. At that time there was an exchange of information concerning a product, Tetradifon ("Tedion"), manufacturing at FMC's Baltimore plant

Page 2

between 1957 and 1970. During the course of the meeting, there were discussions concerning the product, its related manufacturing facilities, disposal of wastes in connection with the manufacture and handling of the product and EPA Region III's letter of June 15, 1983. The meeting concluded with a brief plant tour to identify possible locations for sampling.

On the following day, June 21, 1983, the NUS Corporation representatives returned to the plant and obtained twelve (12) split spoon core samples from various locations throughout the plant site. All samples were split with our plant laboratory personnel.

It is our understanding that NUS will analyze these samples for dioxins as well as for the 129 "priority pollutants" and that the results of such analyses will be available to us in approximately four to six weeks.

If there are questions concerning this letter, or if additional information is required, please advise me (301/355-6400, Ext. 235).

Yours very truly,

D. W. Palmer
Environmental Manager

DWP:ct

cc: Elkins W. Dahle, Jr.
City of Baltimore
Health Department
Bureau of Industrial Hygiene
111 North Calvert Street
Baltimore, Maryland 21202

Art Caple -
Joseph Stang -
State of Maryland
Office of Environmental Programs
201 W. Preston Street
Baltimore, Maryland 21201

FMC Corporation

Agricultural Chemicals Division
1701 East Patapsco Avenue - Box 1616
Baltimore, Maryland 21203
(301) 355 6400

FMC

July 7, 1983

Mr. Neil Swanson
Environmental Scientist
Waste Enforcement Section
Air and Waste Management Division (3AW22)
United States Environmental Protection Agency
Region III
6th and Walnut Streets
Philadelphia, PA 19106

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

Re: Response to EPA Region III
Inquiry on Possible Formation of Dioxins

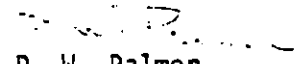
Dear Mr. Swanson:

This letter is to confirm my July 6th telephone conversation with Peter Schaul of EPA Region III.

Because of the difficulty in attempting to locate and review information, some of which dates back twenty-five (25) years, we have requested and Mr. Schaul has agreed to an additional ten (10) days in which to respond to the request for information contained in EPA Region III's letter of June 15, 1983.

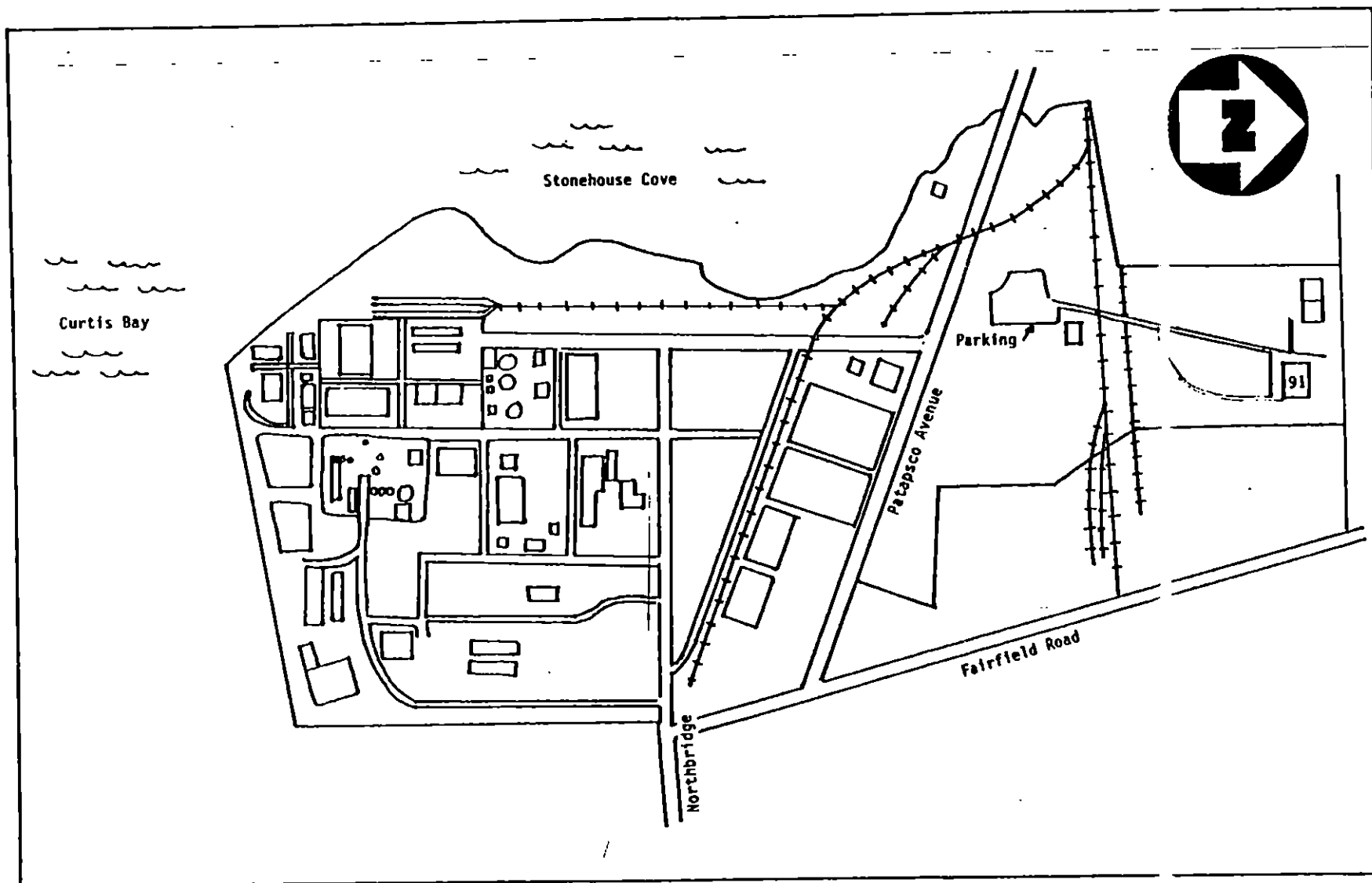
If there are any questions in this regard, please do not hesitate to contact me.

Sincerely yours,


D. W. Palmer
Environmental Manager

DWP:ct

cc: Peter Schaul - EPA Region III

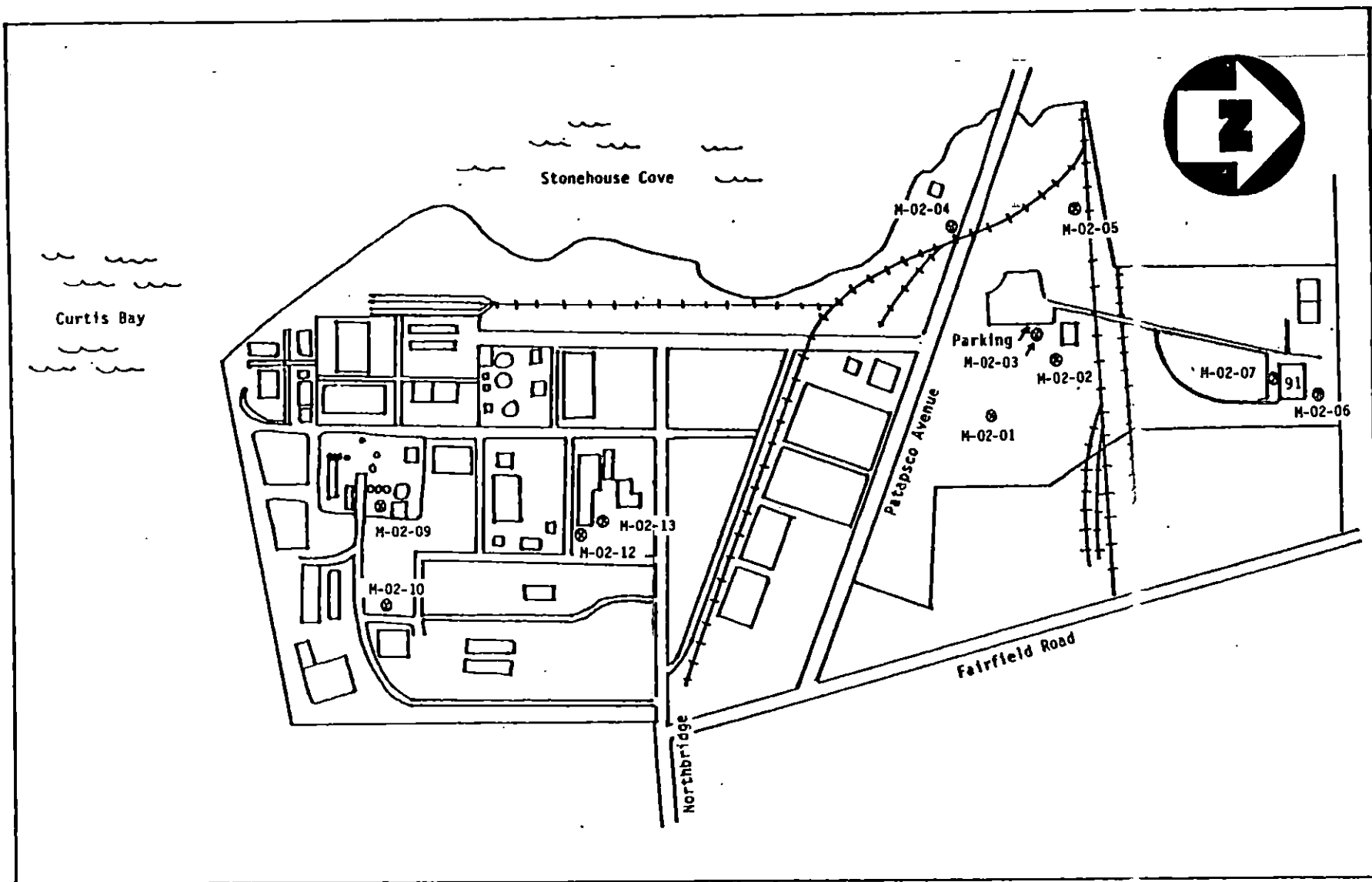


Site Sketch

FMC Corporation, Baltimore, Maryland

NOT TO SCALE



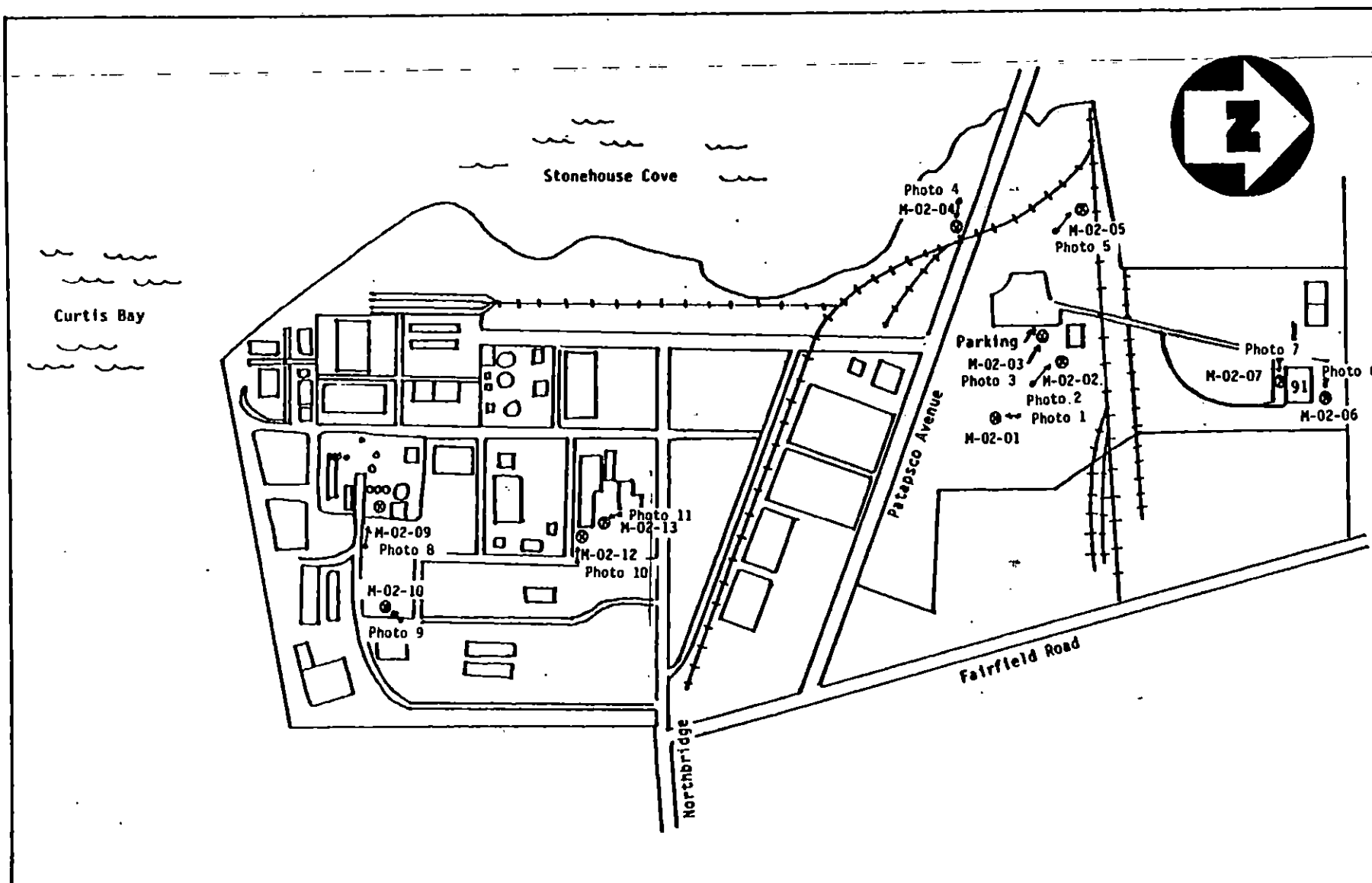


Sample Location Map

FMC Corporation, Baltimore, Maryland

NOT TO SCALE





Photograph Location Sketch

FMC Corporation, Baltimore, Maryland

NOT TO SCALE

APPENDIX D

PROJECT NAME: FMC Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF
DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: 619C (Case 1836)
Contract No.: [Unknown]
Contract Laboratory: Envirodyne
Analytical Protocol: June '83 R.VII, + Memo
Reviewer: R. Sloboda
Review Date: 7/25/83

Applicable Sample No's.: M-02-01, 2, 3, 4, 5, 6, 7, 8,
M-02-09, 10, 12, 13, 14, 14 SPIKE, 15, 16, M-01-01

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		# NA**	NA**	NA**	NA**
Acceptable with exception(s)	✓ 1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- | | |
|--|---|
| <input checked="" type="checkbox"/> DATA COMPLETENESS | <input checked="" type="checkbox"/> QUALITATIVE REQUIREMENTS |
| <input checked="" type="checkbox"/> BLANK ANALYSIS RESULTS | <input checked="" type="checkbox"/> CALIBRATION STANDARDS |
| <input checked="" type="checkbox"/> SURROGATE SPIKE RESULTS | <input checked="" type="checkbox"/> PERFORMANCE AUDIT RESULTS |
| <input checked="" type="checkbox"/> MATRIX SPIKE RESULTS | |
| <input checked="" type="checkbox"/> DUPLICATE ANALYSIS RESULTS | |

Data review forms are attached for each of the review items indicated above.

Comments: ** Not Analyzed for. Data provided can rule out some, but not all, of the other TCDD isomers.
1 please see surrogate spike results

DATA EVALUATION SCORE CATEGORIES

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiencies are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiencies bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiencies imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST p. 1 of 2

SAMPLE NO.	11-02-01	11-02-02	11-02-03	11-02-04	11-02-05	11-02-06	11-02-07	11-02-08	11-02-09	11-02-10
AB I.D. NO. (FRN No.)	23601	23525	23526	23624	23529	23602	23623	23532	23533	23604
MATRIX	Soil							Solvent C1120410	Soil	
RUN DATE/TIME	7/17/85	7/13/85	7/13/85	7/19/85	7/13/85	7/17/85	7/19/85	7/13/85	7/13/85	7/17/85
INSTRUMENT I.D. NO.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
TABULATED RESULTS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DETECTION LIMITS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
SURROGATE ACCURACY	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION RATIOS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
MID CHROMATOGRAMS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN CHROS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
REANALYSIS LOG	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
3 PT. CALIB. R.F./AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
3 PT. CALIB. MID CHROS.	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11
DAILY CALIB. RF/AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DAILY CALIB. MID CHROS.	7/17/85	7/13/85	7/13/85	7/19/85	7/13/85	7/17/85	7/19/85	7/13/85	7/13/85	7/17/85
ISOMER SEPARATION CHROS.	7/17	7/13	7/13	7/19	7/13	7/17	7/19	7/13	7/13	7/17
STANDARD SOURCE	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
EXTRACTION WT.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CALCULATION VOLUMES	✓									
RTIAL SCAN SPECTRA										
HIGH RESOLUTION DATA										
LAB SPIKE RECOVERY										
LAB DUPLICATE				✓						
LAB BLANK										
PERFORMANCE AUDIT SPL.										
INTER-LAB. DUPLICATE					✓*					
SAMPLE BLANK										
DECON. RINSATE							✓			

* Went to Cal. Anal. under SAS618C.

[No sample 11-02-11]

Assume final volume 50µl, according to protocol.

SAMPLE NO.	17-02-12	17-02-13	17-02-14	17-02-14N	17-02-15	17-02-16	17-01-01	17-02-04b	17-02-04b (S-115)	17-02-04b (S-115)	17-02-04b (S-115)
LAB I.D. NO. (FRN No.)	23620	23621	23622	23530	23541	23542	23547	23625	23545	23549	23549
MATRIX	Soil						Powder	Soil	Unknown	Unknown	Unknown
RUN DATE/TIME	7/19 11:40	7/19 13:01	7/19 14:00	7/19 21:21	7/19 21:08	7/19 22:11	7/14 12:53	7/19 16:05	7/14 11:45	7/13 13:30	
INSTRUMENT I.D. NO.	L	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
TABULATED RESULTS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DETECTION LIMITS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
SURROGATE ACCURACY	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION RATIOS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
MID CHROMATOGRAMS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN CHROS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ANALYSIS LOG	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
3 PT. CALIB. R.F./AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
3 PT. CALIB. MID. CHROS.	7/11	7/11	7/11	7/11/7/11	7/11/7/11	7/11/7/11	7/11	7/11	7/11	7/11	7/11
DAILY CALIB. RF/AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DAILY CALIB. MID. CHROS.	7/19 11:00	7/19 11:00	7/19 11:00	7/19 13:00	7/19 13:00	7/19 13:00	7/14	7/19 11:00	7/14	7/13 13:00	
ISOMER SEPARATION CHROS	7/19	7/19	7/19	7/19	7/19	7/19	7/14	7/19	7/14	7/13	
STANDARD SOURCE	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
EXTRACTION WT.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CALCULATION VOLUMES	✓1	✓1	✓1	✓1	✓1	✓1	✓1	✓1	✓1	✓1	✓1
PARTIAL SCAN SPECTRA					✓						
FT-IR RESOLUTION DATA											
LAB SPIKE RECOVERY				✓							
LAB DUPLICATE								✓			
LAB BLANK									✓	✓	
PERFORMANCE AUDIT SPL.					✓	✓					
INTER-LAB. DUPLICATE							✓				
SAMPLE BLANK		✓									
DECON. RINSATE											

† Went to Cal. Anal. under
SAS GISC.

1 Assume final volume 50 µl,
according to protocol.

Blank Analysis Results

The contaminants found in the blanks are listed below:

FRACTION	TYPE OF BLANK	SAMPLE NO.	SOURCE OF water/soil	CONTAMINANTS (concentration/DL)
Soil	sample soil blank	M-02-14	clean soil	TCDD (ND/ DL 0.46 ug/kg)
Soil	lab soil blank	MB(soils) FRN 23545	Lab	TCDD(ND/DL 0.24 ug/kg)
powder	lab powder blank	MB(Powder) FRN 23548	Lab	TCDD(ND/DL 0.84 ug/kg)
Rinsate	1,1,1-trichloroethane rinsate used during sampling equipment decontamination	M-02-CX8	1,1,1-trichloroethane	TCDD (ND/ DL 0.039 ug/kg rinsate)

COMMENTS: No positive sample results from real samples.

SURROGATE SPIKE RECOVERIES

* Asterisked values are outside of QC limits (for action level)

Surrogate compound name:

37Cl₄2,3,7,8-TCDD

Analytical Fraction:		TCDD											
QC LIMITS:		warning:	80.2-113										
		action:	71.9-122										
		source:											
Matrix	Sample no.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.
soil	M-02-01	111											
	-02	93											
	-03	83											
	-04	102											
	-04D	101											
	-05	85											
	-06	95											
	-07	91											
solvent	-08	95											
soil	-09	92											
	-10	98											
[No sample M-02-11]													
soil	-12	93											
	-13	0*											
	-14	104											
	-14N	93											
	-14N ^a	104											
	-15	94											
	-15 ^a	113											
	-16	95											
	-16 ^a	113											
	Method Blank (soils)	83											
powder	M-01-01	99											
	method Blank (powder)	93											

updated 8/12/25

Source of QC Limits: Ref.1: Data Package

Ref.2: Instructional Guide for Reviewing GC/MS Data, version (11/5/82).

COMMENTS: * Lab note indicates interferences; is being re-extracted and reanalyzed.
The results for M-02-013 indicate that the detection limits for 2,3,7,8-TCDD may be significantly above those required (1pob). (Options A and B) were used on M-02-13, but the internal standard and 2,3,7,8-TCDD concs still had interferences after both cleanups.

Matrix Spike Results (spiked by laboratory)

compound	original sample no.	spiked sample no.	concentration		Percent Recovery	Laboratory Control Limits
			ADDED	RECOVERED		
2,3,7,8-TCDD	M-02-14	M-02-14/N	1.17 ug/kg	1.2 ug/kg	103 %	Not specified
2,3,7,8-TCDD	M-02-14	M-02-14/N*	1.17 ug/kg	1.3 ug/kg	111 %	Not specified

* An asterisk indicates values outside control limits.

Comments: Acceptable recoveries, considering that no positive results were encountered for real samples in this project.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No., / Lab Name	Concentration	Sample No. / Lab Name	conc.	Relative Percent Difference
TC ₁₄ -2,3,7,8-TCDD	intra-lab	m-02-04/ Envirodyne	102 % R	m-02-04/ Envirodyne	101 % R	1 %
2,3,7,8-TCDD	intra-lab	m-02-04/ Envirodyne	ND/DL .21 ug/kg	m-02-04/ Envirodyne	ND/DL .19 ug/kg	0
2,3,7,8-TCDD	interlab	m-02-05/ Envirodyne	ND/DL 0.06 ug/kg	m-02-05/ Cal. Anal.	Not yet available	
2,3,7,8-TCDD	interlab	m-01-01/ Envirodyne	ND/DL 0.20 ug/kg	m-01-01/ Cal. Anal.	Not yet available	

Control limits: Not established. Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Results not yet available for m-02-05 and m-01-01 analyses at California Analytical Laboratories.

Qualitative Requirements

A.1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within 8 hours for all positive sample runs? (Y/N) Yes; however,

Exceptions: Negative results were run only on column which did not separate isomers quite as well, but since all positives re-run on original column, data valid.

B.1. 320/322 Ion Ratio within QC Limits (.67-.87) for all positives? Y/N Yes

Exceptions: None. Only positives were QC samples.

C.1. 320, 322, 324 All maximize together (within 3 sec.)? (Y/N) Yes

Exceptions: None

2. S/N greater than 2.5 for each ion? (Y/N) Yes

Exceptions: None

D. Retention time of surrogates and internal standard same as native acid? (Y/N) Yes

Exceptions: None

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) Yes

Exceptions: None

2. High resolution confirmation? (Y/N) No

Comments: Partial scan good enough

3. Partial scan confirmation? (Y/N) Yes

Ion Ratios: QC Limits: 320/322

.67-.87

Sample: 0.82

320/324

1.58 ± 0.16 (1.42-1.74)

1.80 *

257/259

1.03 ± 0.10

0.95

194/196

1.54 ± 0.15

1.45

160, 161, 194, 196, 257, 259, 320, 322, 324 All present except mass 160 *

Comments: Only outliers are detected within as low as above. The partial scan confirmation is of confident matching quality.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes Exceptions: None

Linearity verified within working range? (R² < 10% RSD) Yes Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes Exceptions: None

Check standard R²'s within $\pm 10\%$ of multilevel calibrations? (Y/N) Yes Exceptions: None

Average R² from calibration used in all calculations? (Y/N) Yes Exceptions: None

Performance Audit Results

Source of performance audit samples: Region III EPA soil; blended by Dr. Kline, U.S. Army, for EMSL-LV
Date prepared: 6/28/83 Shellite (if applicable): NA Matrix: Soil
Interferents Added: None

Reference Analysts: None available at this time. Region III rough guess approximately 2 ppb.

Compound	mean value	number of measurements	standard deviation

Performance Audit Sample Results:

Sample no.:	M-02-15	M-02-16	M-02-15	M-02-16
Compound:	2378	2378	2378	2378
concentration: (ug/kg)	3.3	3.1	3.6	3.4
mean value of audit pair (this batch):	3.35	—	—	—
this lab's preceding mean (last batch):	NE#	NE#	NE#	NE#
control limits for mean (this batch):	NE#	NE#	NE#	NE#
control limits for consecutive outliers:	NE#	NE#	NE#	NE#
relative percent difference	—	—	—	—
(RPD) for (this batch) audit pair:	8.7%	—	9.2%	—
RPD for this lab's last batch:	NE#	NE#	NE#	NE#
control limits for RPD (this batch):	NE#	NE#	NE#	NE#
control limits for RPD consecutive:	NE#	NE#	NE#	NE#

* An asterisk indicates values beyond 1.96 standard deviations from the mean.
* A double asterisk indicates values beyond 2.58 standard deviations from the mean.

Comments: NE# = Not yet established. This result series was the first received for this batch of performance audit material from Region III. Since no positive results were encountered in the real samples, and since performance audit samples were positively detected (and expected to be near detection limit), one can therefore assume valid method performance with all other indicators (such as surrogate recoveries) are in control.

PROJECT NAME: EMC Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF
DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: 1836/SAS 619C
Contract No.: [unknown]
Contract Laboratory: Envirodyne
Analytical Protocol : July '83 R.VII + memo
Reviewer: R. Sloboda
Review Date: 10/25/83

Applicable Sample No's.: M-02-13 (Reanalysis)

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDDs	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable					
Acceptable with exception(s)	✓ 1	Not analyzed			
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- | | |
|--|--|
| <input checked="" type="checkbox"/> DATA COMPLETENESS | <input checked="" type="checkbox"/> QUALITATIVE REQUIREMENTS |
| <input checked="" type="checkbox"/> BLANK ANALYSIS RESULTS | <input checked="" type="checkbox"/> CALIBRATION STANDARDS |
| <input checked="" type="checkbox"/> SURROGATE SPIKE RESULTS | <input type="checkbox"/> PERFORMANCE AUDIT RESULTS |
| <input checked="" type="checkbox"/> MATRIX SPIKE RESULTS | <input checked="" type="checkbox"/> CALCULATION CHECKS |
| <input checked="" type="checkbox"/> DUPLICATE ANALYSIS RESULTS | |

Data review forms are attached for each of the review items indicated above.

Comments: 1 Please see qualitative requirements. Only one sample from site investigation reported positive. Although this sample result suggests the presence of TCDD, the following evidence suggests that TCDD result may be artifactual: (1) 2 other labs got a negative TCDD result, with detection limits of 0.2 and 0.27 ppb. One lab was a high resolution lab. (2) Coeluting 320 and 322 interferences, and isolated 257 interferences were seen in all analyses. The 320/322 ratio was correct for one of the peaks in the mixed analysis which was separated from the TCDD retention window. (3) Conversations with Region III EPA chemist Angelo Casarea, Cal. Analytical chemist/Assistant Paul Taylor, and Battelle Hi-resolution chemist Alfred De Roos indicate that artifactual TCDD results do occasionally occur, although identity of the interferent cannot be determined except perhaps by full or partial scan Reanalysis of the original positive extract after reconstitution and extreme concentration.

DATA EVALUATION SCORE CATEGORIES

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiencies are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiencies bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiencies imply the results are not meaningful.

TCDD: DATA COMPLETENESS CHECKLIST

[illegible]

Blank Analysis Results

The contaminants found in the blanks are listed below:

[illegible]

COMMENTS: Sample was run along with a region VII rerun shipment.
QC data for duplicate spikes and method blanks provided verbally
in a telephone conversation w/ Dr. Earl Hanson on 10/3/83.

(Previous for AD w/ DL = 0.21 ppb per conversation above.)

* Asterisked values are outside of QC limits

Surrogate compound name:

37C4-2378-TCDD

[illegible]

Source of QC Limits: Ref.1: September '83 R.VII protocol
Ref.2:

COMMENTS: Acceptable recovery

Matrix Spike Results (spiked by laboratory)

compound	original sample no	spiked sample no	concentration			RELATIVE RECOVERY	LABORATORY CONTROL LIMITS	EPA CONTROL LIMITS
			ADDED	FOUND	UNSPIKED			
2,3,7,8-TCDD	?	?	1.0 ppb					

* An asterisk indicates values outside control limits.

Comments: 10/3 Conversation w/ Dr. Earl Hanson: Mean of 2 spikes was 72% Relative Recovery. Sample was run along with a Region VII shipment.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. / Lab Name	Concentration	Sample No. / Lab Name	conc.	Relative Percent Difference
2,3,7,8-TCDF	interlab	mo2-13/ Envirodyne	1.04 ppb	mo2-13/ Bittell	ND/CL	
2,3,7,8-TCDD	interlab	split of mo2-13/ MEAD	ND/CL 0.20 ppb	split of mo2-13/ Envirodyne	0.22 ppb	200%
2,3,7,8-TCDD	intra lab	split of mo2-13/ MEAD	ND/CL 0.20 ppb	split of mo2-13/ MEAD	0.04 ppb	200%
					0.74%	0%

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: (Region VII samples run in ^{Envirodyne} sample batch. QC samples run in duplicate, and 5.8 and 5.9 ppb found in these analyses.)
Envirodyne was only lab to obtain a positive result for this sample.

Bittell sample was same sample as Envirodyne, EPA Sample # mo2-13. MEAD's sample was a split of the same sample, but sample was homogenized before splitting.

Qualitative Requirements

A.1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within
→ 8 hours to all positive sample runs? (Y/N) Yes

Exceptions: None

B.1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives?

Y/N No Exceptions 0.66923 is slightly below 0.67.

However, positive results have been seen before which are near the borderline of QC criteria.

C.1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) Yes

Exceptions None

2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

D. Retention time of surrogates and internal standard same as native TCDD?
(Y/N) Yes Exceptions None

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) No Exceptions: None

★ 2. High resolution confirmation? (Y/N) Yes Comments High resolution results indicate TCDD not present with a detection limit of 0.27 pgob. However, a separate extraction was performed, so if sample not homogeneous this does not invalidate result for low resolution.

3. Partial scan confirmation? (Y/N) No

→ Ion Ratios: QC Limits:

320/322

320/324

257/259

194/196

160, 161, 194, 196, 257, 259, 320, 322, 324

Comments Only one positive reported for this site investigation.

Although no reason has been established the same sample analyzed

by Hi-Resolution did not show TCDD. In addition, a split sample did not

show the presence of dioxin with a detection limit of 0.2 pgob at another lab.

(The sample was blended in the field before splitting.) Since 320 and 322 interference were seen, it is possible that chromatographic interference produced an artifactual result.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes
 Exceptions: None

Linearity verified within working range? (RRF \leq 10% RSD) Yes (6.9% RSD)
 Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes
 Exceptions: None

Check standard RRF's within $\pm 10\%$ of multilevel calibrations? (Y/N) Not applicable.
 Exceptions: multilevel standard within 8 hours previous to sample run.

Average RRF from calibration used in all calculations? (Y/N) Yes
 Exceptions: None

CALIBRATION LOG

EQUIVALENT PPE LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2,3,7,8-TCDD 3,7-Cl ₄ -2,3,7,8-TCDD		ISOMER STD. CHECK STD or MULTILEVEL
25		7377	8/25 17:35	0.75	1.25	multi-level
5		7378	8/25 18:11	0.80	1.31	" "
1		7379	8/25 18:53	0.89	1.28	" "
		7380	8/25 19:26	< 25% Variability		Isomer Std.

Calculation check for positive results:

m-02-13: $\frac{(87 + 130) \cdot 25}{(259 + 323) \cdot 10.4} \cdot 0.8233 = 1.04 \text{ pob} \checkmark$

Low level standard: FRN 7379: $RF = \frac{(1076 + 1388) \cdot 25}{(3034 + 3540) \cdot 10} = 0.8897 \checkmark$

PROJECT NAME: FMC Agricultural
TDD NO: F3-5356-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF GC/MS
HIGH RESOLUTION DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: Not EPA Funded

Applicable Sample No's.: 13,228 [FMC Split]

Contract No.: " " "

M-02-13, M-02-14, M-02-16, M-02-14N

Contract Laboratory: Battelle

Analytical Protocol: EPA R-111/Hi-Res. Adapted

Reviewer: R. Sloboda

Review Date:

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		Not analyzed for			
Acceptable with exception(s)	✓ 1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- | | |
|--|---|
| <input checked="" type="checkbox"/> DATA COMPLETENESS | <input checked="" type="checkbox"/> QUALITATIVE REQUIREMENTS |
| <input checked="" type="checkbox"/> BLANK ANALYSIS RESULTS | <input checked="" type="checkbox"/> CALIBRATION STANDARDS |
| <input checked="" type="checkbox"/> SURROGATE SPIKE RESULTS | <input checked="" type="checkbox"/> PERFORMANCE AUDIT RESULTS |
| <input checked="" type="checkbox"/> MATRIX SPIKE RESULTS | <input checked="" type="checkbox"/> CALCULATION CHECKS |
| <input checked="" type="checkbox"/> DUPLICATE ANALYSIS RESULTS | |

Data review forms are attached for each of the review items indicated above.

Comments: ¹ Please see calculation checks. Detection limit for M-02-13 can be more accurately stated as 0.27 pgb. Results suggest Envedure results may be artifactual, or that sample was inhomogeneous. Split sample was field homogenized before being split from M-02-13. (M-02-13 sample sent to this lab (Battelle) from Envedure, & was blended in the field, too.)

DATA EVALUATION SCORE CATEGORIES

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiencies are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiencies bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiencies imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO.	13228	M02-13	M02-14	M02-16	M02-14N	M8	M02-13			
LAB I.D. NO.	—	—	219907	219911	219908	219910	219913			
MATRIX	Soil									
RUN DATE/TIME	*	*	10/19	10/19	10/19	10/19	11/19			
INSTRUMENT I.D. NO.	*	*	VG-7070H	HRMS	Resolution	±30 m/z	at 300 amu	*	*	
TABULATED RESULTS	*	*	✓	✓	✓	✓	✓			
DETECTION LIMITS	*	*	✓	✓	✓	✓	✓			
SURROGATE ACCURACY	✓	*	✓	✓	✓	✓	✓			
ION AREAS	*	*	✓	✓	✓	✓	✓			
ION RATIOS	*	*	✓	✓	✓	✓	✓			
MID CHROMATOGRAMS	*	*	✓	✓	✓	✓	✓			
PREVIOUS RUN AREAS										
PREVIOUS RUN CHROS.										
REANALYSIS LOG										
3 PT. CALIB. R.F./AMTS.			✓	✓	✓	✓	✓			
3 PT. CALIB. MID CHROS.			✓	✓	✓	✓	✓			
DAILY CALIB. RF/AMTS.			✓	✓	✓	✓	✓			
DAILY CALIB. MID CHROS.			✓	✓	✓	✓	✓	→	219915	
ISOMER SEPARATION CHROS.			✓	✓	✓	✓	✓	→	219916	
STANDARD SOURCE			✓	✓	✓	✓	✓			
EXTRACTION WT.	10.8	10.8	10.9	10.4	10.7	10.0	1.0			
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓			
CALCULATION VOLUMES	✓	✓	✓	✓	✓	✓	✓			
PARTIAL SCAN SPECTRA										
HIGH RESOLUTION DATA	✓	✓	✓	✓	✓	✓	✓			
LAB SPIKE RECOVERY					✓					
LAB DUPLICATE										
LAB BLANK						✓				
PERFORMANCE AUDIT SPL.				✓						
INTER-LAB. DUPLICATE										
SAMPLE BLANK										
DECON. RINSATE										

* = Unable to analyze
due to precipitate, so 1.0g reextracted

** per telephone conversation w/ Dr. DeRosier on 11/14/83.

Blank Analysis Results

The contaminants found in the blanks are listed below:

[illegible]

COMMENTS: Lab claims contamination due to V_2 % carryover from previous run. (However, previous run data was not included in package.)
No effect on validity of data.

* Asterisked values are outside of QC limits

Surrogate
compound name:

37C1-2378-700

Source of QC Limits: Ref.1: September '83 Revision of Region VIII dioxin protocol
Ref.2:

COMMENTS: ^{net. 2.} Acceptable relative recovery.

Matrix Spike Results (spiked by laboratory)

compound	original sample no	spiked sample no	concentration			RELATIVE RECOVERY	LABORATORY CONTROL LIMITS	EPA CONTROL LIMITS
			ADDED	FOUND	UNSPIKED			
2,3,7,8-TCDD	M02-14	M02-14N	1.87	1.58	ND	84%	Not estab	26/136

* An asterisk indicates values outside control limits.

Comments: Acceptable relative recovery.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. /Lab Name	Concentration	Sample No. /Lab Name	conc.	Relative Percent Difference
2,3,7,8-TCDD	interlab	M02-13 Battelle	ND/PL 0.27 ppb	M02-13 MEAD	ND/PL 0.20	0%
2,3,7,8-TCDD	intra lab	M02-13 MEAD	ND/PL 0.70 ppb	M02-13 MEAD	ND/PL 0.20	0%
2,3,7,8-TCDD	interlab	M02-13 Battelle	ND/PL 0.27 ppb	M02-13 Envirodyne	1.04 ppb	200%

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Battelle attempted to analyze both Mead and Envirodyne samples at 10 grams initially. When precipitation of extracts occurred, a 1g aliquot of Envirodyne (EM) sample was analyzed. There was no successful intra-lab duplicate analysis done by Battelle. Envirodyne was the only lab which obtained a positive result for TCDD in soil.

Qualitative Requirements

A.1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) Yes

Exceptions: None

B.1. 320/322 Ion Ratio within QC Limits (67-87) for all positives?

Y/N Yes Exceptions: Only positives were spike and

performance audit sample.

C.1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) Yes

Exceptions: None. Difficult to determine since retention times not printed on chromatograms. Judged by peak width versus apex separation.

2. S/N. greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

D. Retention time of surrogates and internal standard same as native TCD? (Y/N) Yes Exceptions: Same as (C.1.) above.

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) Yes Exceptions: _____

2. High resolution confirmation? (Y/N) Yes Comments: All analyses were performed by Hi-Resolution GC/MS. Verbal conversation with Dr. Fred DeRoes on 11/14 indicate Resolution at mass 320 was about 30000 or greater.

3. Partial scan confirmation? (Y/N) No

→ Ion Ratios: QC Limits:

320/322 _____

320/324 _____

257/259 _____

194/196 _____

160, 161, 194, 196, 257, 259, 320, 322, 324

Comments: Partial scan confirmation could perhaps be performed on Environyne extract to determine what artifact caused the apparent TCD peak in their low resolution analysis of MOZ-13. Extract may no longer be viable, though, so success is not guaranteed.

• Resolution was good enough to distinguish between 1 sulfur and 2 oxygen in MW of 320 ions

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes

Exceptions: Multi-level calibration run below instead of after check standard !!!
However since sample of interest was NB, no effect on validity of data.

Linearity verified within working range? (RRF < 10% RSD) Yes

Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes

Exceptions: None

Check standard RRF's within $\pm 10\%$ of multilevel calibrations? (Y/N) Yes

Exceptions: None

Average RRF from calibration used in all calculations? (Y/N) Yes

Exceptions: None

CALIBRATION LOG

EQUIVALENT PPB LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2,3,7,8-TCDD 3,7,8-TCDF 2,3,7,8-TCDF		ISOMER STD. CHECK STD. or MULTILEVEL
1.0	VG 7070H HRMS	320525	10/25	1.01	1.35	MULTI-LEVEL
1.0		320526		1.00	1.32	
1.0		320527		0.99	1.26	
S		320528		0.89	1.24	
S		320529		0.92	1.24	
S		320530		0.94	1.24	
25		320531		0.91	1.17	
25		320601		0.95	1.17	
25		320602	↓	1.01	1.17	↓
1.0	↓	219915	10/19	0.96	1.21	check std.

Performance Audit Results

Perf. Audit Batch ID: 3.1

Source and Preparation: Region VII EPA soil, Blended by Univ. Nevada for EMSL-LV

Date Prepared: 6/25/83

Analyte and Matrix: 2,3,7,8-TCDD

Interferents added: None

Reference Analysis Results (Received to date):

P.A. Batch ID: 3.1

Analyte: 2,3,7,8-TCDD

SAS (Sample Batch): 619C 619C 619C 629C 629C 630C 631C 640C 641C 642C 643C 656C 657C 691C

Date Analysis: 8/16 7/13-19 7/13-14 7/14 7/14 8/1-2 8/17 8/4-8 8/8 8/27-28 8/16 8/10 8/5-8 7/31-31

LABORATORY: C V V W W V C W W C C/T C V B

SAMPLE NO: M0105 M0215 M0215 M0316 M0316 M0413 P0226 W0212 W0209 W0110 W0418 P0135 P0323 P0527

RESULT: 3.5 3.3 3.6 3.07 2.86 5.3 3.89 2.85 2.35 2.24 3.42 3.8 4.8 3.75

SAMPLE NO: M0106 M0216 M0216 M0317 M0414 P0227 W0213 W0310 W0111 W0418 P0135 P0324

RESULT: 3.6 3.1 3.4 3.27 5.4 3.86 3.01 2.64 2.19 2.73 2.9 4.1

MEAN: 3.55 3.35 3.07 5.35 3.875 2.93 2.50 2.25 3.075 3.35 4.45 3.75

DIFFERENCE: 0.10 0.20 0.20 0.10 0.03 0.16 0.29 0.05 0.69 0.90 0.70

ANALYTE/Perf. Audit Batch: 2,3,7,8-TCDD in 3.1

STATISTIC MEASURED: individual results

Number of values: 26

mean: 3.5

standard deviation: 0.8

Performance Audit Sample Results:

Performance Audit Batch I.D.: 3.1

Sample no.: M0216

compound: 2,3,7,8-TCDD

concentration:

mean value of audit pair (this batch): 3.47

this lab's preceding mean (last batch): 3.75

(2.58σ) control limits for mean (this batch): within limits

(1.96σ) control limits for conservative outliers: within limits

Audit Pair difference: —

(RPD) for (this batch) audit pair: —

RPD for this lab's last batch: —

(2.58σ) control limits for RPD (this batch): —

(1.96σ) control limits for RPD consecutive: —

* An asterisk indicates values beyond 1.96 standard deviations from the mean.

** A double asterisk indicates values beyond 2.58 standard deviations from the mean.

NE = Not established due to insufficient data.

Comments:

Calculation Check:

m-02-16:
(Performance Audit)

$$\frac{(8950.35 + 11533.43)}{(6479.87 + 8253.64)}$$

$$\times \frac{2.5}{10.4 \times 0.96} =$$

calculated value

3.47

reported value

3.47

m-02-13:
(Detection Limit)

$$\frac{11.78}{2798.36}$$

$$\times \frac{2.5}{1.0} \times \frac{2.5}{0.96}$$

$$= 0.274 \text{ ppb D.L.} \neq 0.83 \text{ ppb D.L.}$$

- The discrepancy in the calculated versus reported detection limits arises out of the interpretation of the section of the dioxin protocol which addresses calculation of detection limits. Two chemists, Paul Taylor, PHD, President of Cal. Analytical, and Angelo Carasea, Region VII EPA contributing author of the protocol, agreed with this reviewer in the following interpretation: When interfering peaks greater than 2.5 times the noise level are present in the 237.8-TCDD retention window for both masses 320 and 322, and if one interference is significantly larger than the other, then the detection limit can be stated (conservatively) as 2.5 times the amount calculated by the lower level interfering mass area and the corresponding C13-TCDD mass area. (This is different than the reported detection limit, which was calculated using the sum of the two masses 320 and 322, and which yielded a higher result due to a relatively higher interference at mass 322 versus mass 320.) Mass chromatograms of this sample have been attached to illustrate the observed phenomena.

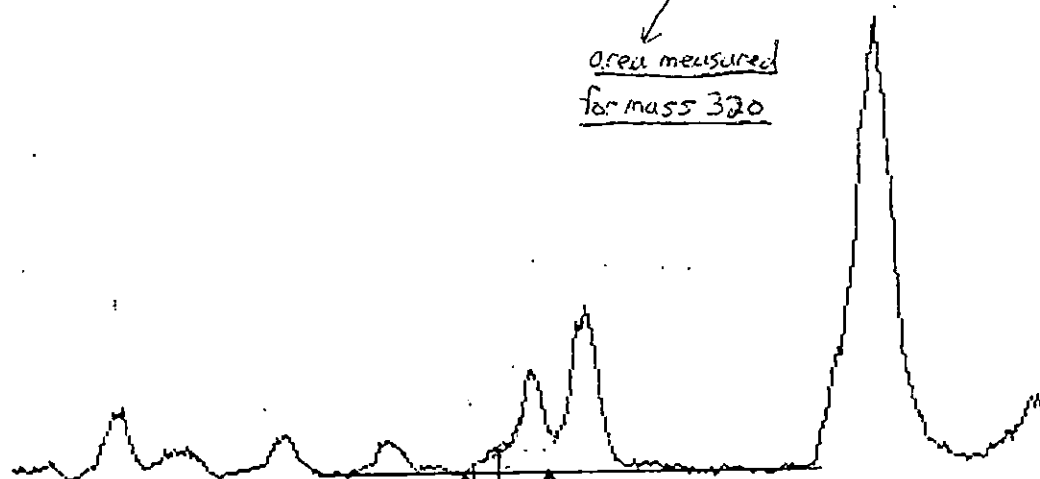
SIR REPORT.
UNCALIBRATED.

FILE - 219913 .SD

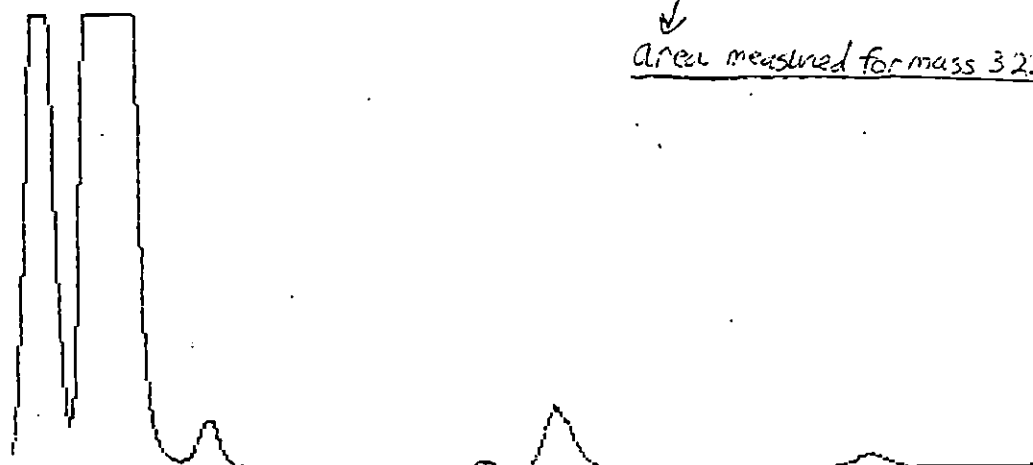
RUN - 1

2UL, 38907-58-8

320 0 HRS 13 MINS 40 SECS 4.8542 11.7820 0.0000



322 0 HRS 13 MINS 39 SECS 51.2444 68.8243 0.0000



measured area
13-2,3,7,8-TCDD Retention Window

FIGURE 5-A. SELECTED ION CURRENT TRACE FOR m/z 320 AND m/z 322 FOR SAMPLE M-02-13

PROJECT NAME: Env. Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF
DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: Not EPA Funded
Contract No.: " " "
Contract Laboratory: Mead Comp/chem
Analytical Protocol: Region VII soil dioxin
Reviewer: R. Sloboda
Review Date: 11/15/83

Applicable Sample No's.: Mead Comp/chem
Sample 13228, which was a split sample of
M-02-13.

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		Not analyzed			
Acceptable with exception(s)	✓ 1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- | | |
|--|--|
| <input checked="" type="checkbox"/> DATA COMPLETENESS ¹ | <input checked="" type="checkbox"/> QUALITATIVE REQUIREMENTS |
| <input type="checkbox"/> BLANK ANALYSIS RESULTS | <input checked="" type="checkbox"/> CALIBRATION STANDARDS |
| <input checked="" type="checkbox"/> SURROGATE SPIKE RESULTS | <input type="checkbox"/> PERFORMANCE AUDIT RESULTS |
| <input type="checkbox"/> MATRIX SPIKE RESULTS | <input checked="" type="checkbox"/> CALCULATION CHECKS |
| <input checked="" type="checkbox"/> DUPLICATE ANALYSIS RESULTS | |

Data review forms are attached for each of the review items indicated above.

Comments: ¹ Data package was not as complete as the Region VII EPA protocol requires. However, essential chromatograms were provided with a brief narrative. Although not all deliverables present, if lab was following Region VII protocol during analysis one can conclude that data rules out the presence of TCDD in the sample aliquot at the reported detection limit of 0.20 ppt. Many interferences were noted in this analysis. Additional cleanup was necessary to remove background. One interfering peak but which had a different retention time than 2,3,7,8-TCDD, had a 320/322 ratio of 0.80, but 257 peak was not seen.

DATA EVALUATION SCORE CATEGORIES

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiencies are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiencies bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiencies imply the results are not meaningful.

TCDD. DATA COMPLETENESS CHECKLIST

SAMPLE NO.	Field Split of Sample	13228 102-13	13228 102-13
LAB I.D. NO.		TH08 5104003	TR08124 A03
MATRIX		SOIL	→
RUN DATE/TIME		10/6 1:37	10/7 13:30
INSTRUMENT I.D. NO.		OWA #3	CLUT #3
TABULATED RESULTS		✓	✓
DETECTION LIMITS		✓	✓
SURROGATE ACCURACY		MS	MS
ION AREAS		✓	✓
ION RATIOS		MS	MS — But can calculate
MID CHROMATOGRAMS		✓	✓
PREVIOUS RUN AREAS		NA	→
PREVIOUS RUN CHROS		NA	→
REANALYSIS LOG		✓	✓
3 PT. CALIB. R.F./AMTS.		MS	MS
3 PT. CALIB. MID CHROS		MS	MS
DAILY CALIB. RF/AMTS.		MS	MS
DAILY CALIB. MID CHROS		10/5/2006 ✓	10/7/2006 ✓
ISOMER SEPARATION CHROS		✓	✓
STANDARD SOURCE		MS	MS
EXTRACTION WT.		MS	MS
CLEANUP METHOD		MS	MS
CALCULATION VOLUMES		✓	✓
PARTIAL SCAN SPECTRA			
HIGH RESOLUTION DATA			
LAB SPIKE RECOVERY			MS
LAB DUPLICATE		✓	✓
LAB BLANK			MS
PERFORMANCE AUDIT SPL.			
INTER-LAB. DUPLICATE			
SAMPLE BLANK			
DECON. RINSATE			
<p>MS = missing data normally required for EPA data package for dioxin.</p>			

Blank Analysis Results

The contaminants found in the blanks are listed below:

[illegible]

COMMENTS: No blank results provided, but not important since
sample results were nondetected.

* Asterisked values are outside of QC limits

37C14-2,378-7CDB

Source of QC Limits: Ref. 1: Region III Soil protocol, September '83 revision.
Ref. 2: 325 181440 115

COMMENTS: * $1 = \text{Area Ratio} = \frac{328}{332+332} = \frac{181440}{19584+218624} = .45$ assuming 25 ng IS, 10 ng Sur.
 RF calculation to 1.14, which is similar to what other labs have reported. Thus, recovery seems reasonable. * $2 = \text{Area Ratio} = \frac{317124}{291456+368064} = .48$ assuming 25 ng IS, 10 ng Sur.
 RF calculation to 1.20, which is similar to what other labs have reported.
 Assuming that regional protocol units were added, it appears surrogate accuracy or relative recovery is reasonable, probably 100% plus or minus 40%.

Matrix Spike Results (spiked by laboratory)

Compound	Original Sample no.	Spiked sample no.	Concentration			Relative Recovery	Laboratory Control Limits	EPA Control Limits
			Added	Found	Unspiked			

* An asterisk indicates values outside control limits.

Comments: Matrix spike results not provided in data package.

Duplicate Analysis Results

Compound	Type of duplicate (Inter/Intra-Lab)	Sample No. / Lab Name	Concentration	Sample No. / Lab Name	Conc.	Relative Percent Difference
Cl ₄ -2,3,7,8-TCDD	intra lab	split of m02-13 Run #1	RF=1.14	split of m02-13 Run #2	RF=1.20	5.1%
2,3,7,8-TCDD	intra lab	split of m02-13 Run #1	ND/DL=0.7ppb	split of m02-13 Run #2	ND/DL=0.2ppb	—
2,3,7,8-TCDD	inter lab	m02-13 Butte Co. Butte Co.	ND/DL=0.27ppb	m02-13 Enbridge Enbridge	1.04ppb	200%

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Enbridge was the only lab to obtain a positive result for this sample. Butte Co. and Enbridge analyzed m02-13. Mead analyzed m02-13 field split. The sample was homogenized in the field (using blender) before splitting. Consequently, MEPC sample should be very similar in composition to m02-13.

Qualitative Requirements

- A. 1. Isomer Specificity Demonstrated in Documentation? (Y/N) No
2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) No
Exceptions: No data provided.
- B. 1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives? (Y/N) NA Exceptions No Positives in this set at this laboratory
- C. 1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) NA
Exceptions No positives in this set, but surrogate and internal standard masses within 3 seconds of each other.
2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None
Could not see height of surrogate in Run #1 Chromatogram, but can tell from labelled area that it was > 2.5 noise since repeats nearby.
- D. Retention time of surrogates and internal standard same as native TDD? (Y/N) Yes Exceptions For sample run #1, I.S. was within 1 second of daily standard. For Run #2, internal standard was within 2 seconds of standard run.
- E. Confirmation Data
1. At least one confirmed per set of 24? (Y/N) NA Exceptions _____
2. High resolution confirmation? (Y/N) Yes Comments High resolution ND run contradicts TDD found in EPA's initial analysis done by Envirotype. Entire result may be artificial or difference may be due to inhomogeneity.
3. Partial scan confirmation? (Y/N) No
→ Ion Ratios: QC Limits: 320/322 _____
320/324 _____
257/259 _____
194/196 _____
160, 161, 194, 196, 257, 259, 320, 322, 324
Comments Data sufficient to rule out the 2,3,7,8-Isomer, provided protocol was followed. Detection limit is roughly verified to one significant figure.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) *N/A*

Exceptions: *N/A data provided*

Linearity verified within working range? (RRE < 10% RSD) *Cannot determine - no data*

Exceptions:

Calibration Check data provided for all sample runs? (Y/N) *Incomplete --*

Exceptions: *Only RIC chromatograms of standards provided*

Check standard RRE's within $\pm 10\%$ of multilevel calibrations? (Y/N) *Cannot determine*

Exceptions:

Average RRE from calibration used in all calculations? (Y/N) *Cannot determine*

Exceptions:

CALIBRATION LOG

EQUIVALENT LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2379-TCDD 37Cl ₄ -2,3,7,8-TCDD	ISOMER STD. CHECK STD or MULTILEVEL
<i>(+ done by Prater?) probably 1ppb</i>	OWA #3	H2831005803	10/5 22:06	?	?
" " "	OWA #3	H5831007A03	11/7 9:04	?	?
					Check Std.
					check std.

• Calculation Check: Rough Estimate to Verify Detection Limit

Run #2: $\rightarrow \frac{(672 + 1120)}{(33248 + 41952)} \cdot \frac{2.5}{100} \cdot \frac{2.5}{0.60} = 0.25$ ppb detection limit calculated estimate
 ≈ 0.30 detection limit reported.

↑ estimated sample weight ↑ estimated RRF ↑ *Correct to one significant figure since RRF should be greater than 0.50.*

Run #1: \rightarrow Cannot verify detection limit because chromatogram not to scale for measuring noise at mass 320, the lower noise ion.